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Title: Final Report Helium Sample Loop Monitoring System

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Final Report
Helium Sample Loop Monitoring System

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C-CDE was requested to assist with a gas analysis system for the Northstar helium flow loop experimental design. The system was requested due to concerns that the lubricating oils in the blower unit could potentially contaminate the helium loop piping system. In proposed operational conditions in the final design of the loop with the production of tritium, there are concerns of isotopic exchange and forming tritiated hydrocarbons followed by the need to address de-contamination and containment of these compounds. Following discussion with loop designers, it was determined that a real-time stream analysis system would be developed as opposed to batch gas sampling. It was felt that a Residual Gas Analyzer (RGA) could be incorporated as the detection method. The RGA operates at vacuum conditions. However, due to the 400 psig operating pressure this choice caused design issues due to pressure safety requirements. The report below details the final design of the system to address this issue and bench-top testing of the unit prior to delivery to the experiment loop system at TA-53. In addition, two samples were collected from the loop during the testing to confirm RGA results.

The goal of the design was to be able to collect “real-time” measurements of the helium gas stream and monitor for light gases and hydrocarbons that could concentrate over time from the lubricating oil degradation. The focus was to monitor for methane (C1) in the gas stream. In addition higher hydrocarbons (>C2) could also be qualitatively identified with the RGA. Due to the possibility of complex gas mixtures the system was also designed to capture gas samples that could be quantitatively analyzed with gas chromatography to separate the potential gas mixtures.

The final design of the system is shown in Figure 1 and was built on a movable test stand in order to be integrated into a helium flow loop prototype system. The enabling technology to allow the monitoring of gases from a high pressure process with a mass spectrometer operating under low pressure (vacuum) conditions is a high-pressure, adjustable leak valve. The component list is documented in Table 1. The system was designed in compliance with LANL’s Engineering Standards Manual, Chapter 17, and Pressure Safety.

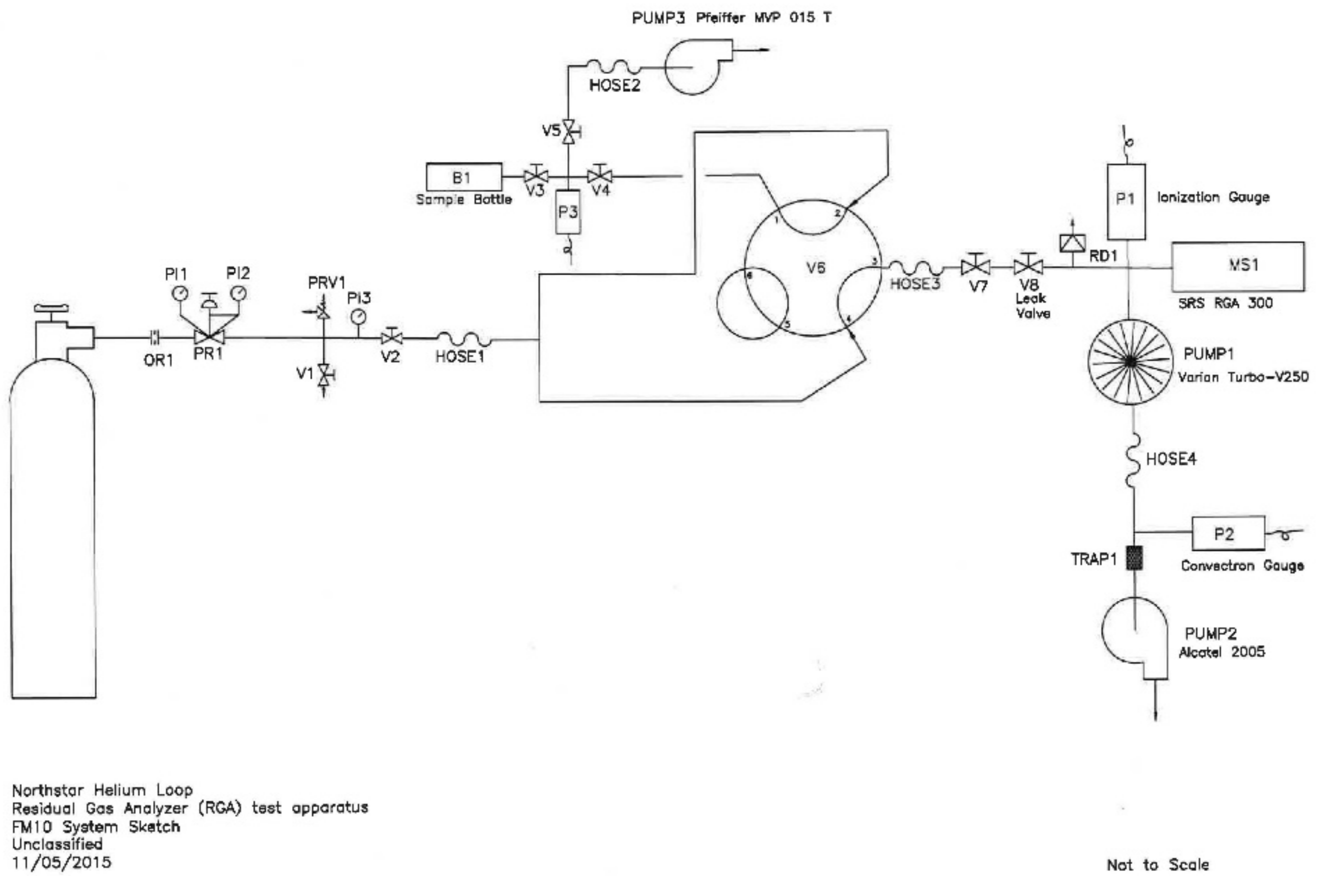

Figure 1 RGA Sampling Manifold

Prototype Northstar Loop RGA					11/16/2015						
ID Number	System Name	TA	BLDG	ROOM	ComponentID		Manufacturer	ModelNumber	Material	MAWP	AddComment
	Northstar Loop RGA	35	0085W	119	Description	COMPONENT ID NUMBER			316 SS, Brass, etc	(psig)	
					Regulator	PR1	Fisher/Victor Eqt. Co	HPS4-3000-580-4F	Brass	3000	Flow Coefficient Cv=0.103
					Gauge, press. ind.	PI1	US Gauge	BU-2581-AM	Brass	4000	
						PI2	US Gauge	BU-2581-AM	Brass	4000	
						PI3	Wika	9768653	316 SS	600	
					Valve, Manual	V1	Swagelok	SS-1RM4	316 SS	5000	
						V2	Swagelok	SS-4BW-V51	316 SS	1000	
						V3	Swagelok	SS-4BG-V51	316 SS	1000	
						V4	Swagelok	SS-4BK-V51	316 SS	1000	
						V5	Swagelok	SS-4BW-V51	316 SS	1000	
						V7	Swagelok	SS-4BK-V51-VS	316 SS	1000	
					Valve, 6 way	V6	Swagelok	SS-43Y6FS2	316 SS	2500	
					Valve, Leak	V8	Agilent	951-5106	316 SS	500	
					Valve, Pressure Relief	PRV1	Swagelok	SS-4R3A5-450	Brass	450	gass supply PRV, yellow spring
					Orifice, Flow reducing	OR1	Swagelok	SS-4-A-RFO-030	316 SS	6600	0.030 orifice
					Transducer, Pressure	P3	Ashcroft	K1	316 SS	1000	proof pressure
					Bottle, Sample	B1	Whitey	4CS-TW-10	316 SS	1000	
					Hose, convoluted metal	HOSE1	Swagelok	FM series	316 SS	3100	working pressure
					with SS braid	HOSE2	Swagelok	FM series	316 SS	3100	working pressure
						HOSE3	Swagelok	FM series	316 SS	3100	working pressure
					Transducer, Vacuum	P1	MDC	432026	misc	NA	Excluded Chap. 17, Ionization Gauge
						P2	Granville Phillips	275071	misc	NA	Excluded Chap. 17, Convectron Gauge
					Hose, vacuum	HOSE4	MDC	UB-KFRL-150-24F	316 SS	NA	Excluded Chap. 17, vacuum component
					Mass Spectrometer	MS1	SRS	RGA 300	misc	NA	Excluded Chap. 17, vacuum component
					Disk, Rupture	RD1	Accuglass	PBD12-133	316 SS	9	protection for vacuum components
					Pump	PUMP1	Varian	Turbo V-250	misc	NA	Pump Rate=?
						PUMP2	Alcatel/Adixen	2005	misc	NA	Pump Rate=?
						PUMP3	Pfeiffer	MVP 015 T	misc	NA	Pump Rate=?
					Trap, foreline	TRAP1	Leybold	NA	316 SS	NA	Excluded Chap. 17, vacuum component

Table 1 - Component List

The system was designed to allow for continuous monitoring of a gas stream by the RGA through a multiport gas sampling valve. The proof-of-concept system was configured to either flow inert helium gas from the cylinder (depicted on the left side of Figure 1) or switch to a small sample bottle filled with a gas mixture or liquid sample to identify low concentration contaminants in the vapor phase. In the final design, the integration of the helium loop replaces the gas cylinder prior to the valve labeled V2. The RGA was manufactured by SRS with the follow specifications;

Sensitivity (A/Torr)	$2X10^{-4}$ (FC), <200 (EM) User adjustable throughout high voltage range. Measured with N2 @ 28 amu with 1 amu full peak width, 10% height, 70 eV electron energy, 12 eV ion energy, and 1 mA electron emission current
Minimum detectable Partial Pressure	$5 X 10^{-11}$ Torr (FC), $5 X 10^{-14}$ Torr (EM), Measured with N2 @28 amu with 1 amu full peak width, 10% height, 70 eV electron energy, 12 eV ion energy, and 1mA electron emission current.
Operating range	10^{-4} Torr to UHV (FC), 10^{-6} Torr to UHV (EM)

FC – Faraday Cup collection
EM – Electron Multiplier collection
UHV – Ultra high vacuum <10^{-6} Torr

To address the performance of the system the following procedure was developed using calibrated gas standards.

4% Gas Standard Mix

- Gas Standard Mix line connected
- evacuate manifold to closed Gas Standard Mix cylinder
- isolate manifold from pump
- expand Gas Standard Mix into manifold
- evacuate manifold
- repeat above fill and evacuate steps two additional times
- fill manifold with Gas Standard Mix to approximately 8 psig
- slowly open leak valve
- average 50 scans at RGA pressure of 8e-08 Torr

Results from this test are shown below in Figure 2, all gases but hydrogen were identified due to the 3 m/z lower set point restriction of the RGA. Carbon Monoxide and Nitrogen, having the same molecular weight 28 m/z, are identified at the same peak. Methane and doubly charge oxygen can also be associated at the same peak, 16 m/z.

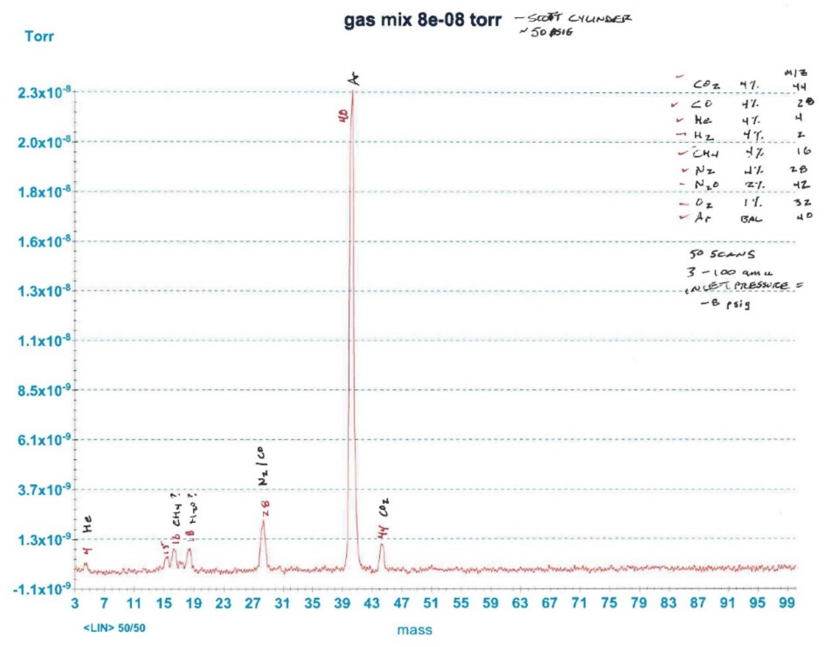

Figure 2 RGA results from 4% Gas Standard Mix

10 ppm Gas Standard Mix

- Regulator manifold, CGA590, had 14 psi PRV, so 10 psi was maximum gas delivery pressure.
- Gas Standard Mix line connected
- evacuate manifold to closed Gas Standard Mix cylinder
- isolate manifold from pump
- expand Gas Standard Mix into manifold
- evacuate manifold
- repeat above fill and evacuate steps two additional times
- fill manifold with Gas Standard Mix to approximately 8 psig
- slowly open leak valve
- average 50 scans at a pressure of 5e-07 Torr (did not see any of the minor peaks at 5e-08 Torr)

The RGA results are presented in Figure 3, full scan and Figure 4 magnification of m/z scale of interest.

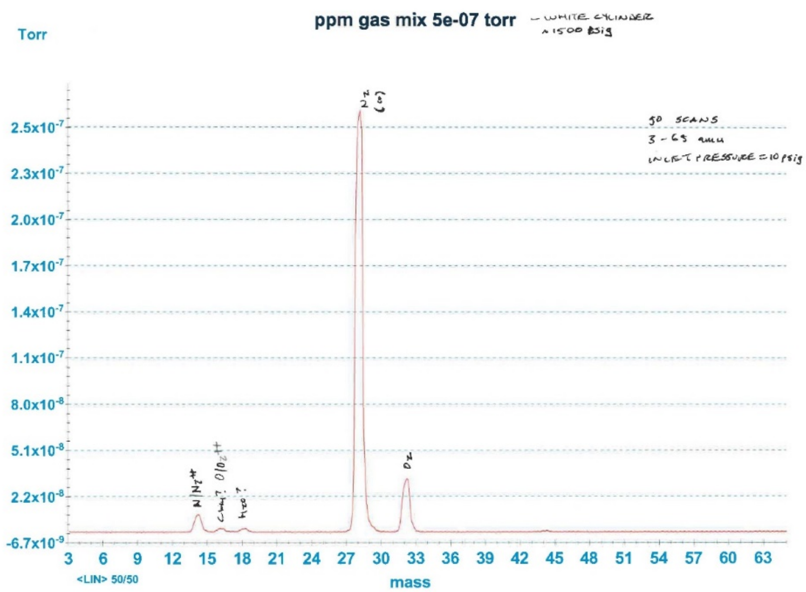

Figure 3 10ppm RGA results

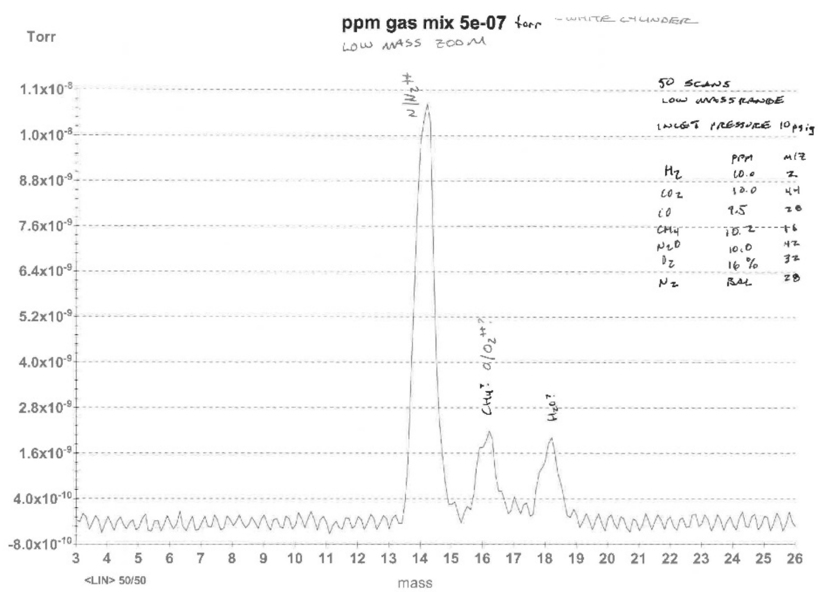

Figure 4 - Magnification for methane identification, 16 m/z

In the 10 ppm mix as stated above, m/z 16 could be CH4 or due to monatomic and/or doubly-charged oxygen (see reference mass spectrum below with peak at m/z 16, Figure 5). At 10 ppm, CH4, with O2 present, cannot be definitively identified with the SRS RGA. However, the experiment will run in a pure helium stream where oxygen is not expected and it is anticipated that interference of oxygen in the identification of the mass spectral peak at m/z 16 as CH4 will not be an issue.

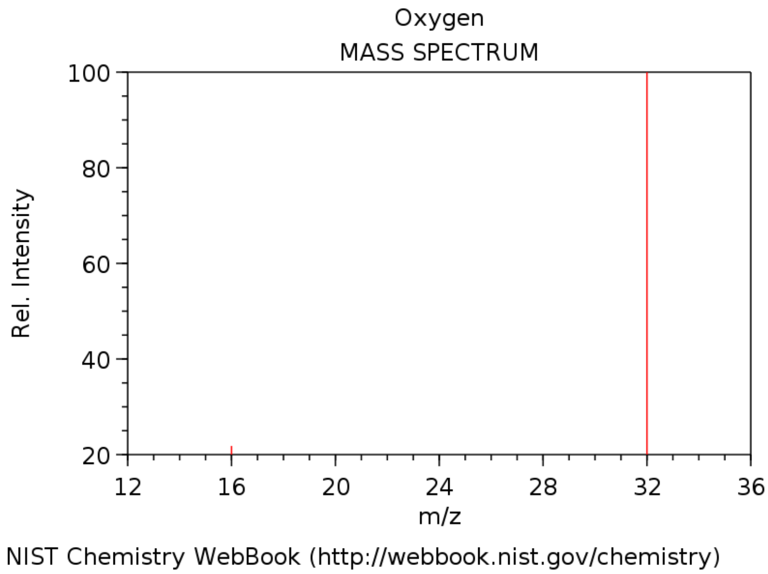

Figure 5 Oxygen mass spectrum showing 16 m/z doubly charged O

The system was also tested with oil samples supplied by the project that are used in the blower. Two oil tests, one with a drop of oil on filter paper under vacuum and the second with 400 psig helium over-pressure were tested. No significant peaks due to compounds in the oil were detected by the RGA over a range of RGA vacuum readings from 6e-8 to 5e-7 Torr for both samples described above. This was believed to be due to the low vapor pressure of the oil at ambient temperature.

Following these laboratory performance tests, the unit was transferred to the TA-53 helium loop experimental site and attached to the system. CDE trained the operators on the hardware and software operations and the system was turned over to the project.

In addition to RGA gas monitoring, two gas samples were pulled from the helium loop on 1/8/16 and 2/8/16 using the manifold system. These samples were analyzed on a gas chromatograph (GC) with a thermal conductivity detector (TCD). The GC was calibrated for carbon dioxide (CO2), carbon monoxide (CO), methane (CH4), hydrogen (H2) and nitric oxide (NO). Results are shown in Table 2 below. Nitrogen and oxygen were also seen in both chromatograms but were not quantitated.

Gas Component	Results 1/8/2016 mole %	Results 2/8/2016 mole %
Carbon Dioxide	0.001	0.011
Methane	ND	ND

Table 2 - Gas Sample results from Helium Loop

RGA monitoring during the Helium Loop runs was outside of the CDE work scope and the above data can be used for a comparison for results found during the experiments. From gas chromatograph data collected, there was not an appearance of significant oil degradation as manifested by the production of CO_2 or CH_4 gas from the oil during operation of the high pressure helium loop.